Date: February 22, 2000 (Rev. # 2) SOP No. ISSI-VBI70-09

Title: METAL SPECIATION AND QUANTIFICATION OF PERLITE

APPROVALS:

Author: ISSI Consulting Group, Inc. Original Date: 9-10-99

SYNOPSIS: A standardized method for speciating metals and perlite particles in solid samples is described. Equipment operating conditions, sample preparation and handling, and statistical equations for data analysis and presentation are included.

REVIEWS:

TEAM MEMBER	SIGNATURE/TITLE	DATE
USEPA Region 8	Brent Lowell /KPM	2/22/00
ISSI Consulting Group, Inc.	WS Bratton	2/22/00

Revision Date	Reason for Revision
11-3-99	Incorporated a methodology for perlite analysis by both optical microscopy and electron microprobe analysis. Provided a method for collecting photomicrographs.
2-22-00	Revised Analysis Logsheet (Figure 11-1) to include k-ratios instead of concentration factors for elements used in the calibration procedure.

1.0 OBJECTIVES

The objectives of this Standard Operating Procedure (SOP) are to specify the proper methodologies and protocols to be used during metal speciation of various solid samples (including tailings, slags, sediments, dross, bag house dusts, and paint), residential soils and dusts for metals. The metal speciation data generated from this SOP may be used to assess the solid samples as each phase relates to risk. Parameters to be characterized during the speciation analyses include particle size, associations, stoichiometry, frequency of occurrence of metal-bearing forms and relative mass of metal-bearing forms. In addition, aliquots of solid samples can be analyzed separately for perlite, using the same methodology. Perlite particles are counted and sized based on the elemental constituents of each particle. This electron microprobe (EMP) technique, instrument operation protocols and sample preparation to be used during implementation of the Metals Speciation SOP are discussed in the following sections.

2.0 BACKGROUND

To date, numerous forms (phases) of metal-bearing particles have been identified in soils from various environments within western mining districts (Table 2-1) (Emmons et al., 1927; Drexler, 1992; Davis et al., 1993; Ruby et al., 1994; CDM, 1994; WESTON, 1995). This listing does not preclude the identification of other metal-bearing forms, but only serves as an initial point of reference. Many of these forms are minerals with varying metal concentrations (e.g., lead phosphate, iron-lead oxide, and slag). Since limited thermodynamic information is available for many of these phases and equilibrium conditions are rarely found in soil environments, the identity of the mineral class (e.g., lead phosphate) will be sufficient and exact stoichiometry is not necessary.

It may be important to know the particle-size distribution of metal-bearing forms in order to assess potential risk. It is believed that particles less than 250 microns (μm) are most available for human ingestion and/or inhalation (Bornschein, et al., 1987). For this study, the largest dimension of any one metal-bearing form will be measured and the frequency of occurrence weighted by that dimension. Although not routinely performed, particle area can be determined. It has been shown (CDM, 1994) that data collected on particle area produces similar results. These measurements add a considerable amount of time to the procedure and limit the total number of particles or samples that can be observed in a study.

Mineral association may have profound effects on the ability for solubilization. For example, if a lead-bearing form in one sample is predominantly found within quartz grains while in another sample it is free in the sample matrix, the two samples are likely

to pose significantly different risk levels to human health. Therefore, associations of concern include the following:

1) free or liberated
2) inclusions within a second phase
3) cementing
4) alteration rims

3.0 SAMPLE SELECTION

Samples will be selected and handled according to the procedure described in the Project Plan.

4.0 SCHEDULE

A schedule for completion of projects performed under this Metals Speciation SOP will be provided in writing or verbally to the contractor along with monthly reporting requirements if large projects are performed. These schedules are based on an aggressive analytical program designed to ensure that the metals speciation analyses are completed in a timely period. Monthly reports are expected to reflect schedule status.

5.0 INSTRUMENTATION

Metal Speciation

Speciation analyses will be conducted at the Laboratory for Environmental and Geological Studies (LEGS) at the University of Colorado, Boulder or other comparable facilities. Primary equipment used for this work will include:

Electron Microprobe (JEOL 8600) equipped with four wavelength spectrometers, energy dispersive spectrometer (EDS), BEI detector and the TN-5600 data processing system. RJ Lee ZEPPELIN and DATALINK hardware may be used for image storage and processing. An LEDC spectrometer crystal for carbon and LDE-1 crystal for oxygen analyses will be used.

Qualitative Perlite Analysis

Identification of perlite morphology will be performed with an optical microscope that utilizes polarized transmitted light.

6.0 PRECISION AND ACCURACY

6.1 Precision

Metal Speciation

The precision of the data generated by the manual particle light microscopy (PLM) particle count and by the "EMP point count" will be evaluated by preparing a graph that compares the original result with the duplicate result.

In general, detectable concentrations for these variable, metal-bearing forms will be determined by performing "peak counts" on the appropriate wavelength spectrometer. Average concentrations will then be used for further calculations. Data on specific gravity will be collected from referenced databases or estimated based on similar compounds. A minimum of two peak counts per sample will be performed on non-stoichiometric phases, as appropriate (that is, if they exist in that sample).

The precision of quantitative elemental analysis will be evaluated based on sample duplicates analyzed at a frequency of 10%.

Perlite

The precision of the data generated by the manual particle light microscopy (PLM) particle count will be evaluated based on sample duplicates analyzed at a frequency of 10%.

6.2 Accuracy

The accuracy of the analyses will be estimated based on a number of methods, depending on the source of the data. Data generated by the "EMP point count" will be evaluated statistically based on the methods of Mosimann (1965) at the 95% confidence level on the frequency data following Equation 1.

$$E_{0.95} = 2P(100-P)/N \qquad \text{(Eq. 1)}$$

Where: $E_{0.95}$ = Probable error at the 95% confidence level

P = Percentage of N of an individual metal-bearing phase based on percent length frequency

N = Total number of metal-bearing grains counted

7.0 PERSONNEL RESPONSIBILITY

The analyst(s) will carefully read this SOP prior to any sample examination.

It is the responsibility of the laboratory supervisor and designates to ensure that these procedures are followed, to examine quality assurance (QA) samples and replicate standards, and to check EDS and WDS calibrations. The laboratory supervisor will collect results, ensure they are in proper format, and deliver them to the contractor.

Monthly reports summarizing all progress, with a list of samples speciated to date with data analyses sheets (DAS), will be submitted each month.

It is also the responsibility of the laboratory supervisor to notify the contractor representative of any problems encountered in the sample analysis process.

8.0 SAMPLE PREPARATION

Grain mounts (1.5 inches in diameter) or polished sections of each sample will be prepared using air-cured epoxy. This grain mounting technique is appropriate for both the electron microprobe (EMP) and polarized light microscopy (PLM) analyses. However, separate splits for the two analyses must be prepared. The grain mounting is performed as follows:

1) Log the samples for which polished mounts will be prepared.

2) Inspect all disposable plastic cups, making sure each is clean and dry.

3) Label each "mold" with its corresponding sample number.

4) All samples will be split to produce a homogeneous 1-4 gram sample.

5) Mix epoxy resin and hardener according to manufacturer's directions.

6) Pour 1 gram of sample into mold. Double check to make sure sample numbers on mold and the original sample container match. Pour epoxy into mold to just cover sample grains.

7) Using a new wood stirring stick with each sample, carefully blend epoxy and grains so as to coat all grains with epoxy.

8) Set molds to cure at ROOM TEMPERATURE in a clean, restricted area. Add labels with sample numbers and cover with more epoxy resin. Leave to cure completely at room temperature.

9) One at a time remove each sample from its mold and grind flat the back side of the mount.

10) Use 600 grit wet abrasive paper stretched across a grinding wheel to remove the bottom layer and expose as many mineral grains as possible. Follow with 1000 grit paper.

11) Polish with 15 um oil-based diamond paste on a polishing paper fixed to a lap. Do not use cloth, as the use of paper will minimize relief.

12) Next use 6um diamond polish on a similar lap.

13) Finally, polish the sample with 1um oil-based diamond paste on polishing paper, followed by 0.05 um alumina in water suspension. The quality will be checked after each step. Typical polishing times are 30 minutes for 15 um, 20 minutes for 6 um, 15 minutes for 1 um, and 10 minutes for 0.05 um.

NOTE: use low speed on the polishing laps to avoid "plucking" of sample grains.

14) Samples will be completely cleaned in an ultrasonic cleaner with isopropyl alcohol or similar solvent to remove oil and fingerprints.

15) To ensure that no particles of any metal are being cross-contaminated during sample preparation procedures, a blank (epoxy only) mold will be made every 20^{th} sample (5% of samples) following all of the above procedures. This mold will then be speciated along with the other samples.

16) Each sample must be carbon coated. Once coated, the samples will be stored in a clean, dry environment with the carbon surface protected from scratches or handling.

9.0 PERLITE ANALYSIS USING POLARIZED LIGHT MICROSCOPY

Optical microscopy will be employed to determine the morphology of perlite particles, if present, in each investigative sample. The perlite samples will be mounted on glass thin sections prior to polishing. Perlite particles will be counted under polarized transmitted light in the magnification range of 45-400X. Any particle that appears to be perlite (based on morphology) will be counted, and the number of the perlite particles will be documented on the Perlite DAS (Figure 9-1). The visual identification of perlite particles will be confirmed by EMP, as discussed below.

10.0 GEOCHEMICAL SPECIATION USING ELECTRON MICROPROBE

All investigative samples will also be characterized using EMP analysis to determine the chemical speciation, particle size distribution and frequency for several target metals. These are arsenic, lead, cadmium, zinc, thallium, selenium, indium, antimony, and mercury. Additionally, chemical speciation and particle size distribution for perlite will also be determined. The perlite speciation will be performed to confirm the morphological results obtained using the PLM.

10.1 Instrument Calibration and Standardization

The WDS will have spectrometers calibrated for the metals of concern (As, Pb, Cd, Zn, Tl, Se, Sb, Hg and In), carbon, oxygen and sulfur on the appropriate crystals using mineral standards. The EDS will have a multi-channel analyzer (MCA) calibrated for known peak energy centroids. Calibration will be performed so as to have both low (1.0-3.0 KeV) and high (6.0-9.0 KeV) energy peaks fall within 0.05 KeV of its known centroid.

The magnification marker on the instrument will be checked once a week. This will be performed by following manufacturer instructions or by measurement of commercially available grids or leucite spheres. Size measurements must be within 4 microns of certified values.

Initial calibration verification standards (ICVs) must be analyzed at the beginning of each sample delivery group or once every 48 hours, whichever is more frequent. A set of mineral or glass standards will be run quantitatively for the metals of concern (As, Pb, Cd, Zn, Tl, Se, Sb, Hg and In), sulfur, oxygen and carbon. If elemental quantities of the ICVs do not fall within 5% of certified values for each element, the instrument must be recalibrated prior to analysis of investigative samples.

The metal-bearing forms in these samples will be identified using a combination of EDS, WDS and BEI detection. Once a particle is isolated with the backscatter detector, a 5-second EDS spectra is collected and peaks identified. The count rates for the metals of concern, sulfur, carbon and oxygen can be either visually observed on the wavelength spectrometers or K-ratios calculated.

10.2 Analytical Procedure

A brief visual examination of each sample will be made prior to EMP examination. This examination may help the operator by noting the occurrence of slag and/or organic matter. Standard operating conditions for quantitative and qualitative analyses of metal-bearing forms are given in Table 10-1. However, it is the responsibility of the operator to select the appropriate analytical line (crystal/KeV range) to eliminate peak overlaps and ensure proper identification/quantification of each analyte. Quality control will be maintained by analyzing duplicates at regular intervals (Section 6.1).

The backscattered electron threshold will be adjusted so that all particles containing the element of interest (i.e., both those with low content and high content) can be seen. For particles that have generally high content (i.e., those that are especially bright), it may be necessary to lower the threshold in order to identify sub-regions of the particle that may contain different phases. This procedure will minimize the possibility that metal-bearing minerals may be overlooked during the scanning of the polished grain mount. The scanning will be done manually in a manner similar to that depicted in Figure 10-1. Typically, the magnification used for scanning all samples except for airborne samples will be 40-100X and 300-600X. The last setting will allow the smallest identifiable (1-2 um) phases to be found. Once a candidate particle is identified, then the backscatter image will be optimized to discriminate any different phases that may be making up the particle or defining its association. Identification of the metal-bearing phases will be done using both EDS and WDS on an EMP, with spectrometers peaked at sulfur, oxygen, carbon and the metal of concern (M). The size of each metal-bearing phase will be determined by measuring the longest dimension (in microns).

10.3 Point Counting

Counts are made by traversing each sample from left-to-right and top-to-bottom as illustrated in Figure 10-1. The amount of vertical movement for each traverse would depend on magnification and CRT (cathode-ray tube) size. This movement will be minimized so that NO portion of the sample is missed when the end of a traverse is reached. Two magnification settings generally are used. One ranging from 40-100X and a second from 300-600X. The last setting will allow one to find the smallest identifiable (1-2 micron) phases.

The portion of the sample examined in the second pass, under the higher magnification, will depend on the time available, the number of metal-bearing particles, and the complexity of metal mineralogy. Eight hours will be spent on each sample.

The particle counting goal for the As, Pb, Cd, Sb, Tl, In, Hg and Se analysis is to count the number of particles for each metal type until 8 hours has expired. Zinc particles will also be counted during this 8-hour period. However, only particles having a zinc composition of greater than 2% (mass/mass) will be scanned and analyzed.

Perlite

Particles that are tentatively identified as perlite using PLM will be examined by EPM to identify the major elemental constituents. A particle will be identified as perlite if the elemental composition contains silica, aluminum and potassium in relative amounts such that a spectrum is produced that is similar to the example provided in Figure 10-2.

10.4 Quantitative Analyses

Quantitative analyses are required to establish the elemental stoichiometry (and hence the elemental mass fraction) of the metal-bearing minerals. Quantitative analyses of representative phases will be performed for each of the target elements (As, Pb, Cd, Sb, Tl, In, Se, Zn and Hg).

Results for each phase will be averaged across several representative grains to establish mean values. The range of values may also be depicted as histograms to show the range of metal concentrations measured as well as the presence of one or more populations in terms of metal content. In the latter case, non-parametric statistics may have to be used or the median value has to be established. Photomicrographs and EDS spectra will be printed, as described in Section 11.2. Example EDS spectra for common or important phases are provided as an attachment (Attachment A).

10.5 Associations

The association of the metal-bearing forms will be established from the backscattered electron images. Particular attention will be paid in establishing whether the grains are totally enclosed, encapsulated or liberated. The rinds of metal-bearing grains will be identified. Representative photomicrographs of backscatter electron images establishing the association of the principal metal-bearing forms will be obtained for illustration purposes.

10.6 Source Material Dilutions

In the case where test materials are highly enriched with target metals (e.g., PAX, flue dust), it may be necessary to dilute the sample to facilitate sample characterization. This will be performed at the discretion of the analyst. For samples that are determined to require dilution, clean quartz sand (SiO_2) will be mixed with the sample material. The sand will be certified to be free of target analytes. The quartz sand will be added to an aliquot of the investigative sample, then mixed by turning the sample for a minimum of one hour, or until the sample is fully homogenized. The initial mass of the investigative sample aliquot, and the mass of the quartz addition must be recorded on the Data Analysis Sheet (Figure 10-3).

11.0 DATA RECORDING AND DOCUMENTATION

11.1 Analysis Results

Analysts will record information on the initial calibration for each metal, and the results for each sample (in counts observed for each 8-hr period) on the Analysis Logsheet (Figure 11-1).

Analysts will electronically record data as they are acquired from each sample using the LEGS Microsoft Access® software, which places all data in a spreadsheet file format. An example of the data entry screen is provided in Figure 11-2. An example of the spreadsheet format is provided in Figure 10-3. Data entry fields have been established for numbering the metal-bearing phase particles, their identity, size of longest dimension in microns, along with their association (L = liberated, C= cementing, R = rimming, I = included). The analyst will also summarize his/her observations in the formatted data summary files.

11.2 Photomicrographs and EDS Spectra

Photomicrographs must be taken for each sample, at a rate of 5% (1 photograph per 20 particles counted) for a maximum of 10 photographs per sample, and submitted with the sample results. Particles selected for photography must be recorded on the EMP graph, as well as in the Photomicrograph Logbook (Figure 11-3). Each photo will be assigned a number, starting with 1 and increasing until the project is complete. Any additional photographs will be labeled as "opportunistic" and must be accompanied by a brief explanation of the reason for taking the picture. For each photomicrograph that is taken, an EDS spectrum of each target chemical present in the sample will also be printed. Each spectrum will be assigned a number as follows:

- The same integer used for the corresponding photomicrograph will also be used for the EDS spectrum.

- Each photo will also be assigned a letter ID. Therefore, if 3 spectra are generated for photo #3, the spectrum IDs would be 3A, 3B, and 3C, respectively.

A positive black and white film (Polaroid 52) will be used, and a 128x128 (minimum) binary image in ".tif" format will be developed and stored, using a high resolution scanner. Recorded on each photomicrograph will be a scale bar, magnification, sample identification, date and phase identification. Abbreviations for the identified phases can be used. Examples are listed in Table 10-2. A final list must be submitted with the laboratory report, along with printouts of the corresponding EDS spectra for each particle that was photographed. An example photomicrograph is provided in Figure 11-4.

11.3 Data Storage and Archival

For each day of data acquisition, all electronic files (DAS forms and other sample logbook sheets) will be saved on the hard drives of the local instrument computer and the general laboratory computer. All files will be saved under a directory reserved for the VBI70 project. The files for each analytical batch (each day's run) will be stored in a unique subdirectory. The subdirectories will be named as the analysis data (e.g., 12-01-99). Additionally, the files for each analysis data will be stored on a floppy disk. Floppy disks will be maintained in a single location with limited access.

11.4 Data Reduction and Reporting

The frequency of occurrence and relative metal mass of each metal-bearing form as it is distributed in each sample will be depicted graphically as a frequency bar graph. The particle size distribution of metal-bearing forms will be depicted in a histogram. Size-histograms of each metal-bearing form can be constructed from data in the file.

Data from EMP will be summarized using two methods. The first method is the determination of FREQUENCY OF OCCURRENCE. This is calculated by summing the longest dimension of all the metal-bearing phases observed and then dividing each phase by the total.

Equation 2 will serve as an example of the calculation.

$$F_M \text{ in phase-1} = \frac{\Sigma (PLD)_{\text{phase 1}}}{\Sigma (PLD)_{\text{phase-1}} + \Sigma (PLD)_{\text{phase-2}} + \Sigma (PLD)_{\text{phase-n}}} \quad \text{(Eq. 2)}$$

Where:
F_M = Frequency of occurrence of metal in a single phase.
PLD = An individual particle's longest dimension
$\%F_M$ in phase-1 = F_M in phase-1 * 100

These data thus illustrate which metal-bearing phase(s) are the most commonly observed in the sample or relative volume percent.

The second calculation used in this report is the determination of RELATIVE METAL MASS. These data are calculated by substituting the PLD term in the equation above with the value of M_M. This term is calculated as defined below.

$$M_M = FM * SG * ppm_M \quad \text{(Eq. 3)}$$

Where:
M_M = Mass of metal in a phase
SG = Specific Gravity of a phase
ppm_M = Concentration in ppm of metal in a phase

The advantage in reviewing the RELATIVE METAL MASS determination is that it gives one information as to which metal-bearing phase(s) in a sample are likely to control the total bulk concentration for a metal of interest. For example, PHASE-1 may comprise

98% relative volume of the sample; however, it has a low specific gravity and contains only 1,000 parts per million (ppm) arsenic. PHASE-2 comprised 2% of the sample, has a high specific gravity, and contains 850,000 ppm of arsenic. In this example it is PHASE-2 that is the dominant source of arsenic to the sample.

Finally, a concentration for each phase is calculated. This quantifies the concentration of each metal-bearing phase. This term is calculated as defined below (Eq. 4).

$$ppm_M = M_M * \text{Bulk metal concentration in ppm} \quad \text{(Eq. 4)}$$

12.0 PERSONAL HEALTH AND SAFETY

Each individual operating the KEVEX x-ray fluorescence or electron microprobe instruments will have read the "Radiation Safety Handbook" prepared by the University and follow all State guidelines for operation of X-ray equipment.

Latex gloves and particulate masks will be worn during preparation of sample cups. All material that comes in contact with the samples or used to clean work surface areas will be placed in poly-bags for disposal.

13.0 SAMPLE HANDLING, CUSTODY AND ARCHIVAL

13.1 Sample Handling and Custody

All raw sample material, pucks and thin mounts will be clearly labeled and handled to avoid cross-contamination and damage. These samples and associated chain-of-custody forms will be stored in a location such as a lockable file cabinet having limited access.

13.2 Unused Sample

All raw, unused sample will be returned to the Contractor under chain-of-custody at the completion of the study.

14.0 FINAL REPORT AND DELIVERABLES

14.1 Final Report

A final laboratory report will be prepared for the Contractor. The report will include all EMP data including speciation results, summary tables and figures, EDS spectra, and photomicrographs. A narrative that describes any deviations to this SOP or the

associated Project Plan(s), and the reason for the deviation will be included in the case narrative.

Speciation results will include: 1) a series of tables summarizing frequency of occurrence for each metal phase identified along with a confidence limit; 2) summary histograms of metal phases identified for each waste type; 3) a summary histogram of particle size distribution in each waste type; and 4) a summary of metal phase associations.

14.2 Deliverables

The following deliverables will be submitted to the contractor at the completion of the study:

- Final Laboratory Report
- EDS Spectra
- Photomicrographs
- CD-ROM of all electronic files generated throughout the study (including raw data files, figures, tables, and the final report file) as follows:
  - Sample.geo
  - Sample.xls
  - Sample.wb3
  - Photo#.tif
  - Size.xls
  - Ass.xls
  - Sam.xls
  - Probe.xls

15.0 REFERENCES

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Table 2-1

Metal-Bearing Forms Found Within Western Mining and Smelting Districts

OXIDES

- Lead Oxide
- Manganese (metal) oxide
- Iron (metal) oxide
- Lead (metal) oxide
- Lead molybdenum oxide
- Arsenic (metal) oxide
- Cadmium Oxide
- Copper Oxides
- Zinc Oxide
- Lead Arsenate
- Arsenic Trioxide
- Calcium (metal) oxide

SILICATES

- Slag
- Lead silicate
- Arsenic silicate
- Zinc silicate
- Clays

SULFATES

- Iron (metal) sulfate
- Lead sulfate
- Lead barite
- Zinc Sulfate
- Arsenic sulfate
- Copper sulfate

CARBONATES

- Lead Carbonate
- Zinc Carbonate

PHOSPHATES

- (metal) phosphates

SULFIDES

- Lead sulfide
- Sulfur-containing salts
- Iron-arsenic sulfide
- Zinc sulfide
- Copper sulfides
- Copper-iron sulfide
- Cadmium Sulfide

OTHER

- Native: Lead, Copper, Cadmium, Mercury, Indium, Thallium, Selenium
- Lead/Arsenic/Cadmium/Mercury Chlorides
- Lead paint
- Solder
- Organic lead
- Lead vanadate
- Minor telluride, and bismuth-lead phases

Figure 9-1. Perlite Data Analysis Sheet

Polarized Transmitted Light Microscopy

Date: _____ Analyst: _____

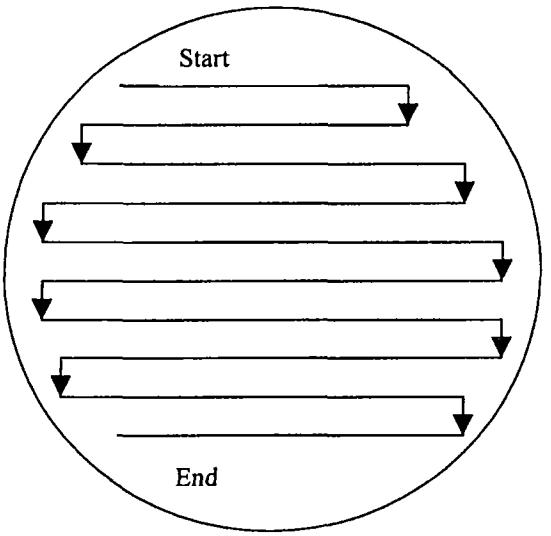
Analytical Sequence #	Sample ID	Magnification	Qualitative Perlite Count (# of particles)	Notes *

* Indicate any required dilutions: initial sample mass plus SiO_2 addition

Logbook Page Reviewed By: _____

Date: _____

Figure 10-1



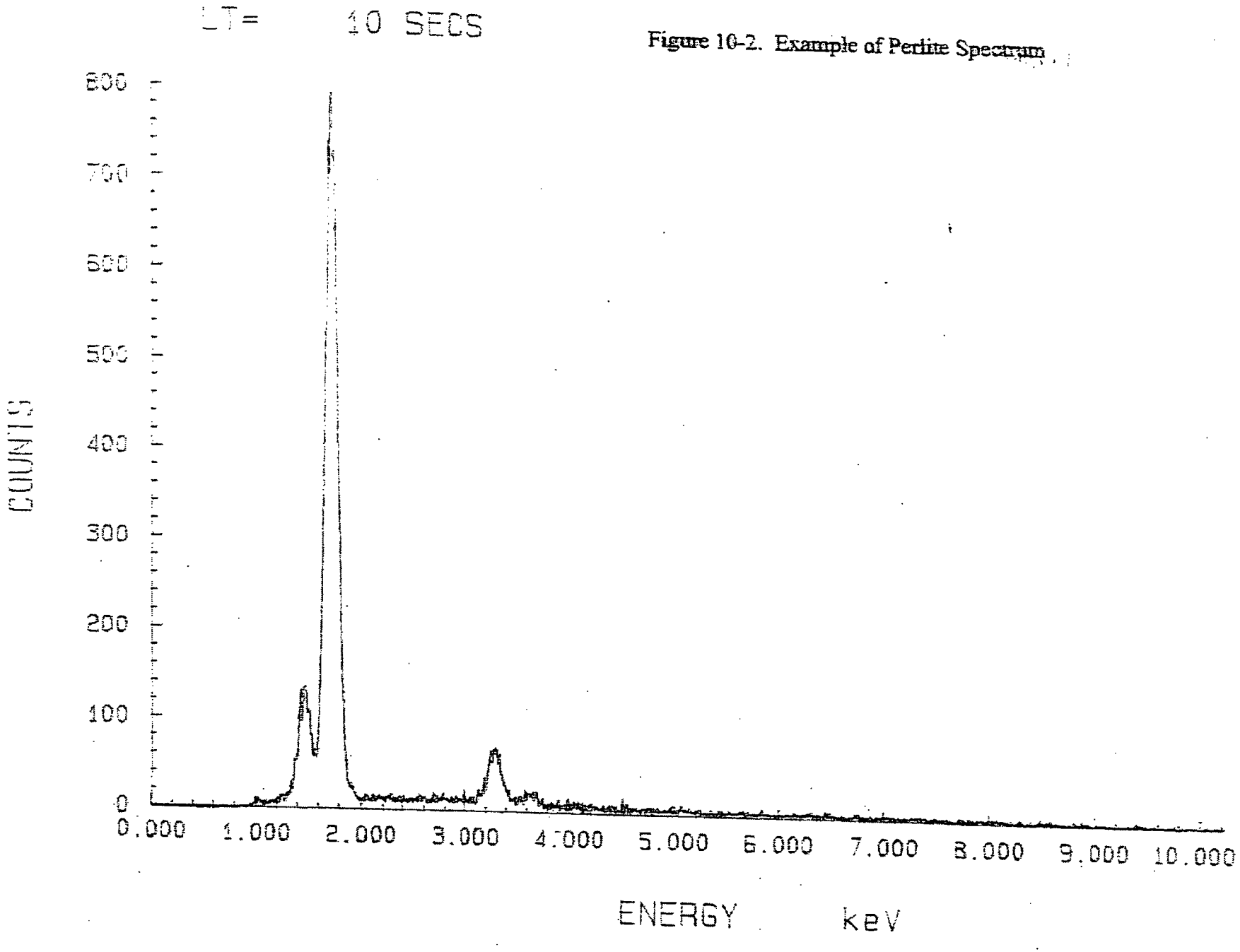


Figure 10-2. Example of Perlite Spectrum

Figure 10-3. Geochemical Speciation Data Analysis Sheet

Analytical Sequence #	Notes *	Sample ID	Size		Liberated	Inclusions	Cemented	Rim
			Length (um)	Area (um)				

* Indicate any required dilutions: initial sample mass plus SiO_2 addition

Logbook Page Reviewed By: ______________________

Date: ______________________

Table 10-1

EMP Standard Operating Conditions

	WDS	EDS
Accelerating Voltage	15 KV	15-20 KV
Beam Size	1-2 microns	1-2 microns
Cup Current	10-30 NanoAmps	10-30 NanoAmps
Ev/Channel	NA	10 or 20
Stage Tilt	NA	Fixed
Working Distance	NA	Fixed
MCA time Constant	NA	7.5-12 microseconds
X-ray lines	S K-alpha PET O K-alpha LDE1 C K-alpha LDEC Zn K-alpha PET As L-alpha TAP Cu K-alpha LIF Cd L-alpha PET Pb M-alpha PET Pb L-alpha LIF In L-alpha PET Tl L-alpha LIF Hg L-alpha LIF Se L-alpha LIF Sb L-alpha PET	S K-alpha 2.31 KeV O K-alpha 0.52 KeV C K-alpha 0.28 KeV Pb M-alpha 2.34 KeV Pb L-alpha 10.5 KeV Zn K-alpha 8.63 KeV Cu K-alpha 8.04 KeV As K-alpha 10.5 KeV As L-alpha 1.28 KeV Cd L-alpha 3.13 KeV In L-alpha 3.28 KeV Tl M-alpha 2.27 KeV Tl L-alpha 10.26 KeV Hg L-alpha 9.98 KeV Hg M-alpha 2.19 KeV Se L-alpha 1.37 KeV Sb L-alpha 3.60 KeV

Table 10-2

Suggested Abbreviation for Photomicrographs

Metal-bearing Phase	Abbreviation
In	In
Tl	Tl
Hg	Hg
Se	Se
Sb	Sb
Lead Sulfide	Ga
Lead Sulfate	Ang
Lead Carbonate	Cer
Mn-(M) Oxide	Mn(M)
Fe-(M) Oxide	Fe(M)
(M)Phosphate	(M)Phos
Fe-(M) Sulfate	Fe(M)Sul
Metal Oxide	(M)O
Pb-Mo Oxide	Wulf
Slag	Slag
Metallic Phase	(M)
Metal Silicate	(M)Si
Solder	Sold
Paint	Pnt
Metal-bearing Organic	(M)(Org)
(M) barite	(M)Bar
Pb arsenate	PbAsO
Pb vanadate	PbVan
As-Sb Oxide	AsSbO
Chalcopyrite	Cp
Sphalerite	Sph
Arsenopyrite	Apy

Figure 11-1. Analysis Logsheet

Analyst: _____ Logbook Page Reviewed By: _____

Magnification Marker [a] _____ Date: _____

Standard ID [b]	Analytical Sequence #	Date/Time [c]	K-ratio	Result [d]	Acceptance Limits	Comments
As	As-1		1		0.95 - 1.05	
Pb	Pb-1		1		0.95 - 1.05	
Cd	Cd-1		1		0.95 - 1.05	
Zn	Zn-1		1		0.95 - 1.05	
Tl	Tl-1		1		0.95 - 1.05	
Se	Se-1		1		0.95 - 1.05	
Sb	Sb-1		1		0.95 - 1.05	
Hg	Hg-1		1		0.95 - 1.05	
In	In-1		1		0.95 - 1.05	
O	O-1		1		0.95 - 1.05	
C	C-1		1		0.95 - 1.05	
S	S-1		1		0.95 - 1.05	

Standard ID [b]	Analytical Sequence #	Date/Time [c]	Sample ID	Phase	Result	Comments

[a] - The magnification marker on the instrument must be checked once a week. If less than one week has elapsed since the last time the magnification marker was calibrated, enter "N/A".
[b] - Initial calibration verification must be performed once every 48 hours.
[c] - Enter time in 24-hour format.
[d] - The calibration standards must be within 5% of the concentration factor to be acceptable.

Figure 11-2

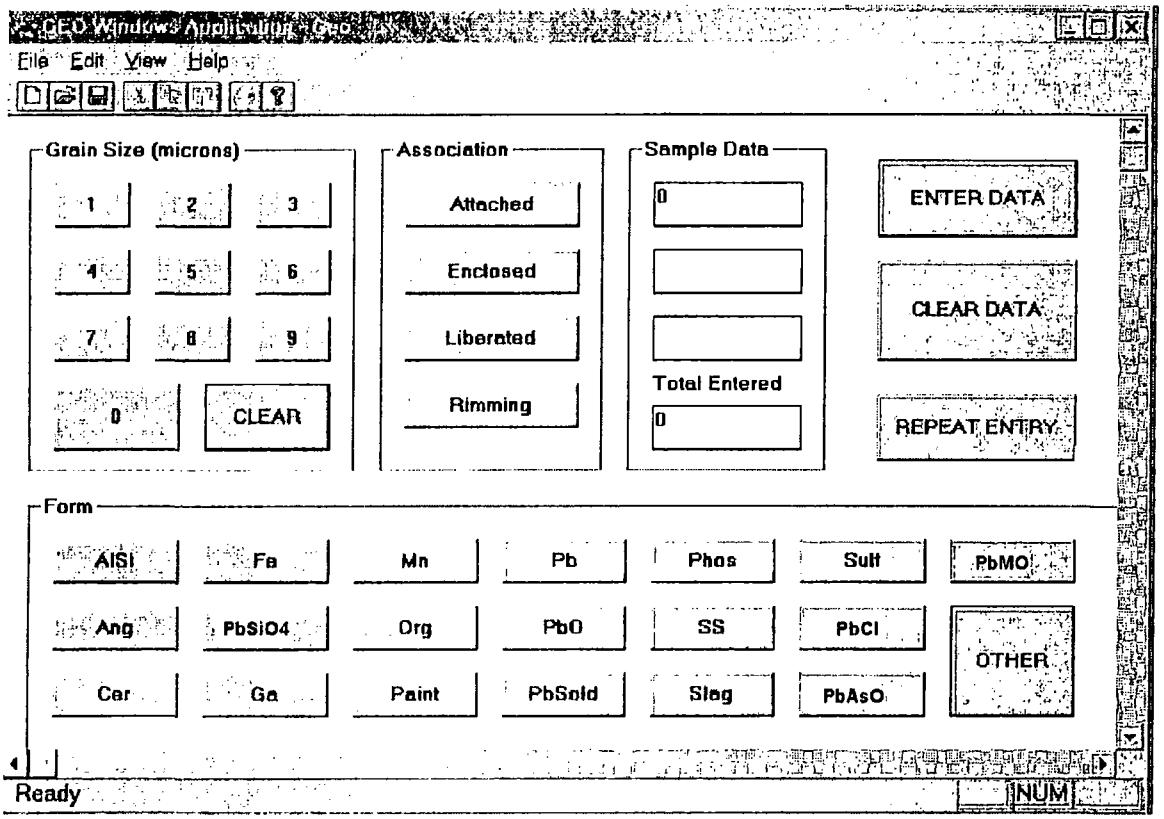

File Edit View Help
Grain Size (microns)
1
2
3
4
5
6
7
8
9
0
CLEAR
Association
Attached
Enclosed
Liberated
Rimming
Sample Data
0
Total Entered
0
ENTER DATA
CLEAR DATA
REPEAT ENTRY
Form
AISI
Fe
Mn
Pb
Phos
Sulf
PbMO
Ang
PbSiO4
Org
PbO
SS
PbCl
OTHER
Car
Ga
Paint
PbSold
Slag
PbAsO
Ready
NUM

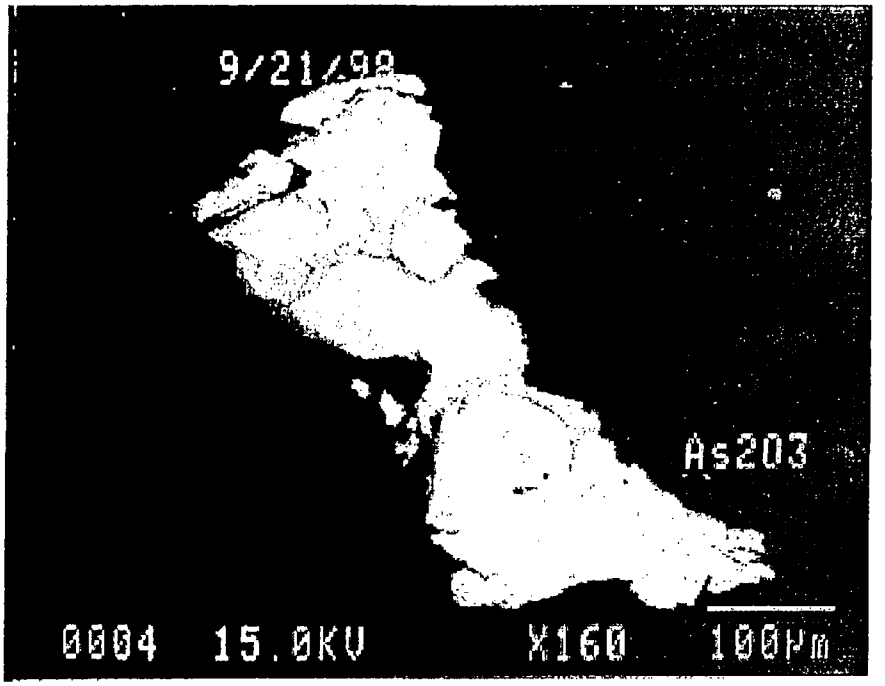
Figure 11-3

Photo Micrograph Logbook				
Sample No.	Particle No. (20, 40, 60, etc.)	Photograph ID	EDS Spectra ID	Comments[a]

a - If discretionary or opportunistic photographs are collected, indicate this and the reason for collection.

Logbook Page Reviewed By: _____ Date: _____

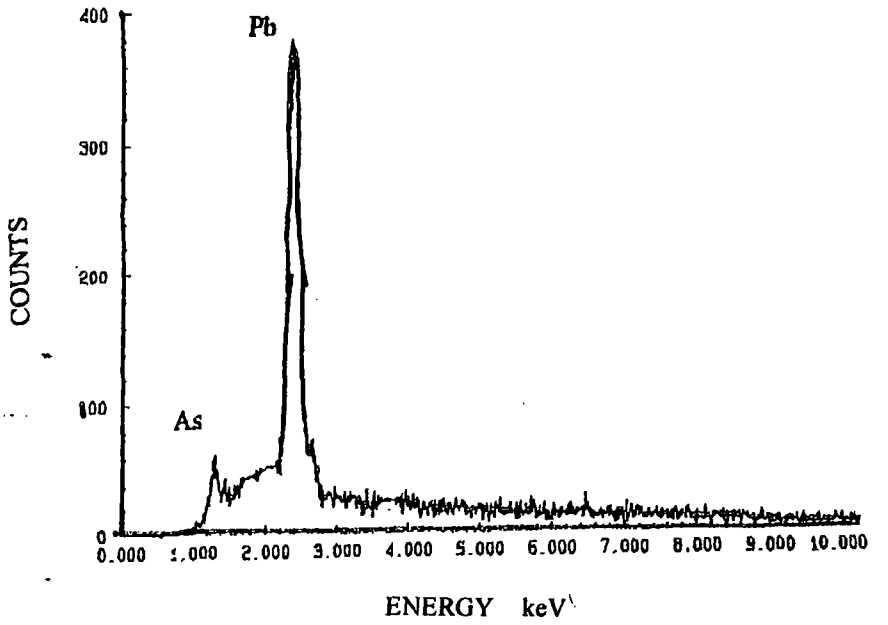
Figure 11-4. Example Photomicrograph



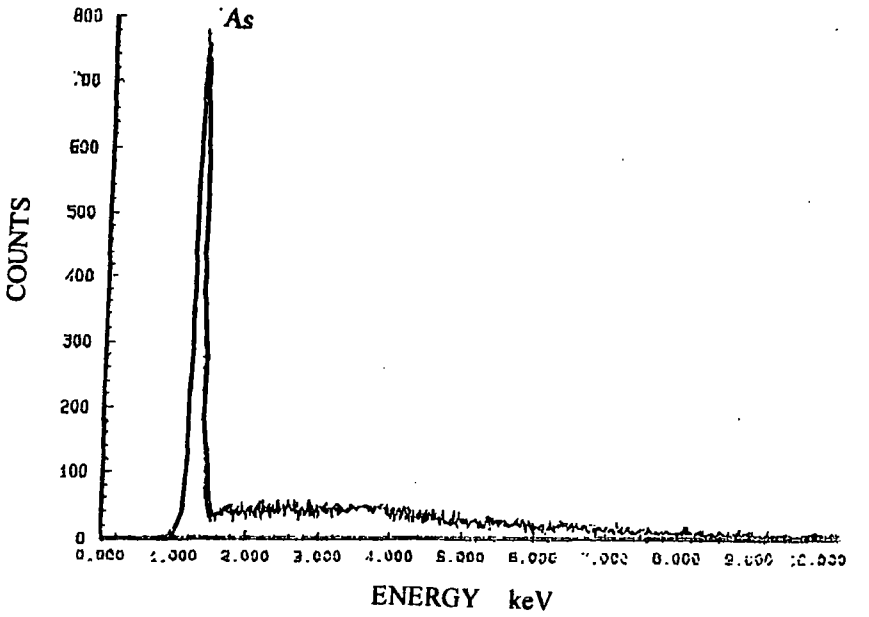
Attachment A

Example Spectra

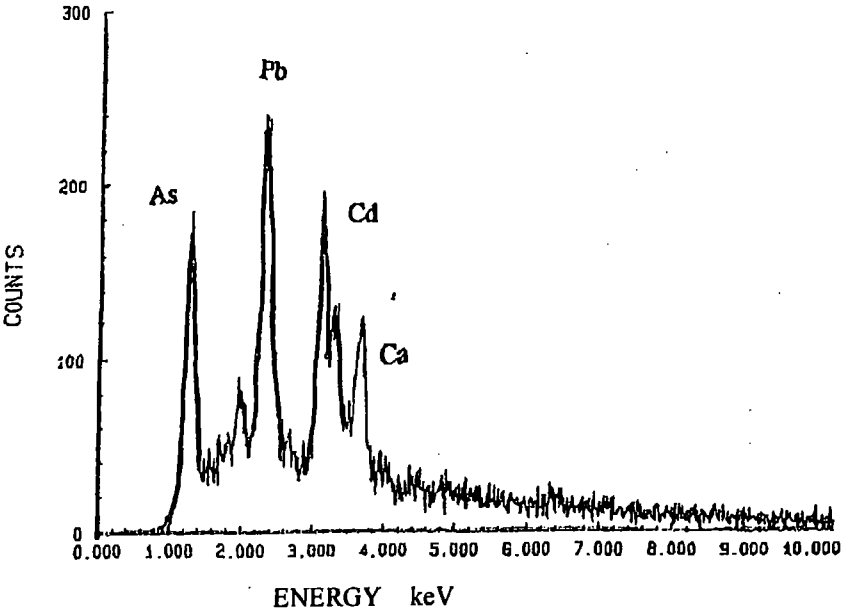
Example Spectrum: Lead Arsenic Oxide



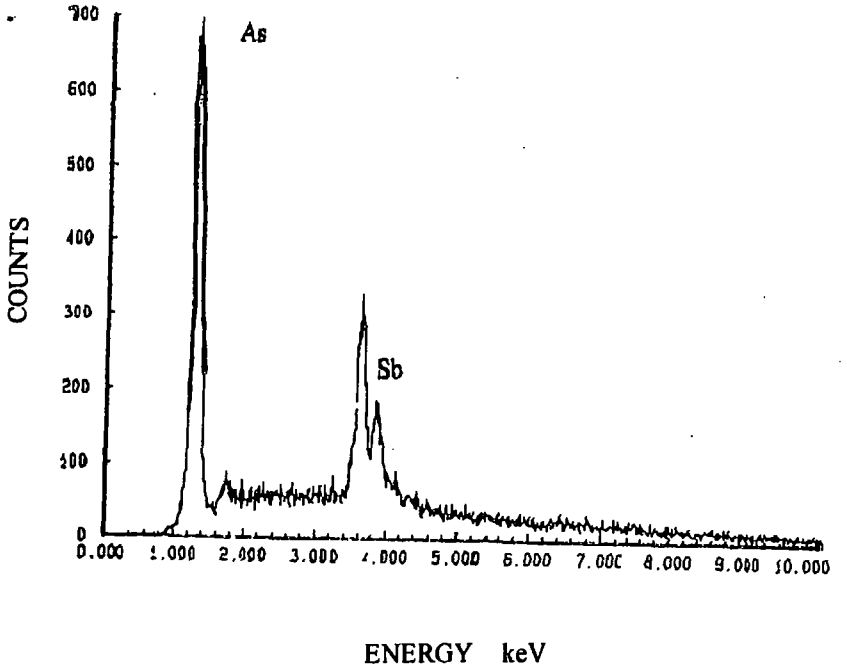
Example Spectrum: Arsenic Trioxide



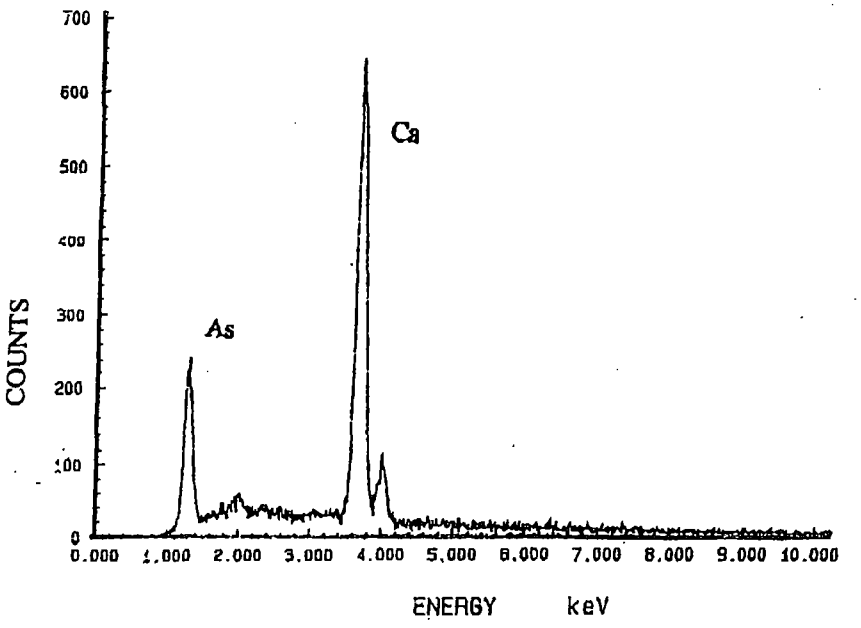
Example Spectrum: Lead/Arsenic Metal Oxide



Example Spectrum: Arsenic Antimony Oxide



Example Spectrum: Arsenic Calcium Oxide



Example Spectrum: Indium